DIRECT BONDING OF GLASS ARTICLES FOR DRAWING

FIELD OF THE INVENTION

[0001] This invention relates to direct bonding of glass. More particularly, the invention relates to methods for direct bonding of a wide variety of glass articles that are subsequently drawn into fibers, rods, sheets, bars or tubes such as optical fiber preforms.

BACKGROUND OF THE INVENTION

[0002] A wide variety of glass articles, such as fibers, sheets, rods, tubes and bars are formed by a glass drawing process in which a glass preform is heated to the softening point of the glass. Tension on a portion of the glass downstream from heated portion of the glass draws the glass into its final form.

[0003] For example, in the manufacture of optical fiber, as shown in Fig. 1, a preform 10, consisting of core surrounded by a cladding is generally arranged vertically in a draw tower 12 so that a portion of the preform 10 is lowered into a furnace 14 that typically heats the preform to temperatures exceeding 2000° C. As the lower end of the preform melts in the furnace, the preform necks down from the original cross-sectional area of the preform to the desired cross-sectional area of a fiber 16. The fiber 16, which is coated in coating apparatus 18, 20 with a polymeric coating, is collected on a spool 22 until the preform 10 is exhausted. After the preform

10 has been exhausted, the draw tower is shut down, until a new preform is loaded into the draw tower.

[0004] This process is inefficient in that shut down of the fiber draw tower results in equipment downtime. One way of improving this inefficiency is by increasing the size of the preform, particularly the diameter. However, a limitation of increasing the size of the preform is the size of optical fiber and other glass preforms which is often limited by the type of equipment utilized to manufacture and consolidate such preforms. In addition, it is difficult to control the optical properties of fibers produced from larger diameter preforms.

[0005] European patent application no. EP 1057793, and United States patent numbers 4,407,667, 6,098,429 and 6,178,779 each disclose methods of joining the ends of optical fiber preforms by heating the preforms to their softening point and fusion bonding the preforms together. EP 1057793 and United States patent no. 6,178,779 disclose using a plasma torch to heat the ends of the preforms together. United States patent no. 6,098,429 states that heating the ends of the preform with a torch may degrade the optical attenuation parameters of optical fiber drawn from such fused preforms. United States patent no. 6,098,429 discloses a method of welding or fusing optical fiber preforms together by using a using a high power laser. Even though the method disclosed in United States patent no. 6,098,429 purportedly represents an improvement, lasers are expensive to implement and pose safety concerns in a manufacturing environment.

[0006] Fusion bonding relates to the process of cleaning two surfaces (glass or metal), bringing the surfaces into contact, and heating close to the softening point of the materials being bonded (to the lower softening temperature for two dissimilar materials), thus forming a welded interface. As noted above, a disadvantage of fusion bonding is that this process typically results in deformation of the two surfaces being bonded due to the flow of softened material. Fusion bonding also tends to result in an interface between the bonded surface that may include bubbles of gas. In addition, fusion bonding typically results in a loss of signal transmitted through the interface for signal transmitting objects such as optical fibers.

[0007] It would be desirable to provide a bonding process for articles that are drawn into fiber, sheets, tubes, rods and bars that does not exhibit the disadvantages of fusion bonding. In particular, in the area of drawing optical fibers, eliminating the problem of softening the ends of the preforms and causing potential attenuation problems in the optical fiber made from the preform would be advantageous. In addition, it would be useful to provide a bonding process that provided high bond strength capable of holding the preforms together during the drawing process, which involves placing the glass preform under tension at high temperatures.

SUMMARY OF INVENTION

[0008] The invention relates to methods of bonding opposing surfaces of glass articles that are subsequently drawn into

sheets, tubes, rods, fibers, bars and ferrules. According to one embodiment, optical fiber preforms are joined at the preform ends, and the composite preform is drawn into an optical fiber waveguide.

[0009] According to another embodiment of the invention, a method of manufacturing a glass article includes providing bonding surfaces on first and second articles, and attaching the bonding surfaces of the first and second articles without an adhesive and at a temperature lower than 1000° C to provide a preform. After the articles are joined to provide a preform, the preform can be drawn to provide a fiber, a rod, a sheet, a bar or a tube. In one such embodiment, the first and second articles are optical fiber preforms and the bonding surfaces are disposed on the ends of the preforms.

[00010] The method may further involve providing a hydrophilic surface on the bonding surface of the first and the second ends of the articles. In another embodiment of the invention, the method may include forming hydrogen bonds between the bonding surfaces of the first and the second articles. Forming hydrogen bonds may include contacting the bonding surfaces of the first and second articles with an acid. In another embodiment, the method may further include providing termination groups on the bonding surfaces of the first and second articles such as -OH, ≡SiOH, $=Si(OH)_2$, $-Si(OH)_3$, $-OSi(OH)_3$, and combinations thereof. Providing these functional groups may further involve contacting the ends of the first

and second articles with a solution having a pH greater than 8. The solution includes a hydroxide such as ammonium hydroxide. According to this embodiment, it is preferred that adsorbed hydroxyl groups are substantially eliminated at the interface between the first and second surfaces by heating the bonding surfaces to a temperature less than the softening or deformation point of the articles. As hydrated surface groups condense under these conditions, water is formed as a byproduct.

[00011] According to another embodiment of the invention, the first and second articles are tubes and the bonding surfaces include sidewalls of the tubes. According to this embodiment, the method is useful for producing fiber ferrules. According to another embodiment, the first and second articles include a polarizing glass containing elongated crystals.

[00012] Another embodiment of the invention relates to a method of forming an optical fiber comprising the steps of bonding the end surfaces of at least two optical fiber preforms without an adhesive and at a temperature less than the softening or deformation temperature of the preforms to provide a blank and drawing optical fiber from the blank. According to this embodiment, the method involves of providing termination groups, preferably, hydroxyl termination groups, on the end surfaces of the preforms. According to another embodiment, the invention may further include heating the end surfaces of the preforms such that absorbed water molecules are driven from the surface and the adsorbed hydroxyl groups

remain on the end surfaces of the preforms. The method may also include forming a covalent bond between the preforms.

[00013] The invention provides a simple, low temperature, and reliable bonding method that provides bond strength capable of surviving high drawing temperatures. Bonding can occur at temperatures lower than the softening or deformation temperature of the glass, and in some cases lower than 100° C. Additional advantages of the invention will be set forth in the following detailed description. It is to be understood that both the foregoing general description and the following detailed description are exemplary and are intended to provide further explanation of the invention as claimed.

BRIEF DESCRIPTION OF THE DRAWINGS

[00014] FIG. 1 is a diagram of an prior art optical fiber draw apparatus;

[00015] FIGS. 2a-2d are diagrams showing the steps of bonding two optical fiber preforms;

[00016] FIG. 3a is a diagram of a prior art method for drawing a sheet or bar of glass;

[00017] FIG. 3b is a diagram of a method of drawing a sheet or bar of glass according to the present invention; and

[00018] FIGS. 4a-4d are diagrams showing a method of drawing a dual ferrule.

DETAILED DESCRIPTION

[00019] According to the present invention, various methods can be utilized to directly bond opposing surfaces of at least

two glass articles together prior to drawing the article into a sheet, a rod, a tube, a bar or a fiber. As used herein, the terms "direct bonding" and "direct bond" means that bonding between two surfaces is achieved at the atomic or molecular level, no additional material exists between the bonding surfaces such as adhesives, and the surfaces are bonded without the assistance of fusion of the surfaces by heating. As used herein, the terms "fusion" or "fusion bonding" refers to processes that involve heating the bonding surfaces and/or the material adjacent the bonding surfaces to the softening or deformation temperature of the articles bonded. The methods of the present invention do not involve the use of adhesives or fusion bonding to bond the opposing surfaces together. Instead, the present invention utilizes methods that involve the utilization of forming a direct bond between the surfaces without high temperatures that soften the glass material to the point of deformation or the softening point, which typically results in an interface that is not optically clear. The present invention provides a bonding method that provides an impermeable, optically clear seal, meaning that there is essentially zero distortion of light passing between the interface of the bonded surfaces. Acceptable methods include, but are not limited to, , wringing, chemical bonding, and vacuum bonding. The formation of a direct bond between two glass or metal surfaces allows for an impermeable seal that has the same inherent physical properties as the bulk material surfaces being bonded.

[00020] Wringing refers to a process of bonding glass surfaces in which adsorbed surface groups are removed from active bonds on a surface by heating the parts to temperatures typically above 600° C but below the softening point of the glass. Adsorbed water and organics will vaporize and the results surface sites become "active." At such a temperature or after cooling in a clean, low humidity environment, surfaces can be placed in contact at which point covalent bonds spontaneously form between "active" bonds on each surface. This is similar to vacuum bonding, except the surface is activated by temperature rather than by a strong vacuum.

[00021] Vacuum bonding involves bringing two clean surfaces into contact in a high vacuum, thus forming a bond. Provided that the surfaces are flat and clean, a high vacuum removes adsorbed water and hydrocarbons from the surface while preventing the adsorption of such species. Surfaces can be cleaved in the vacuum, processed and cleaned before being placed in the vacuum, or cleaned in the vacuum via ion milling or other plasma techniques.

[00022] Within the microelectronics field, vacuum bonding has been developed for sealing of such materials as single crystal silicon, thermal oxide SiO_2 grown on Si, and various metals, as described in United States patent number 6,153,495. Coefficient of thermal expansions (CTE) mismatch between materials is not an issue because the process can be applied

at room temperature. Because polished wafers are thin and typically non-flat due to the Twyman effect, special fixturing can be used to apply pressure evenly across the entire wafer surface to generate appropriate contact.

[00023] Another type of bonding process that may be utilized according to the present invention involves chemical bonding. The formation of a chemical bond between two glass or metal surfaces allows for an impermeable seal that has the same inherent physical properties as the bulk material being bonded. In literature, low-temperature bonding technology has been reported for bonding soda-lime-silicate glass and for crystalline quartz (see, e.g., A. Sayah, D. Solignac, T. Cueni, "Development of novel low temperature bonding technologies for microchip chemical analysis applications," Sensors and Actuators, 84 (2000) pp. 103-108 and P. Rangsten, O. Vallin, K. Hermansson, Y. Backlund, "Quartz-to-Quartz Direct bonding," J. Electrochemical Society, V. 146, N. 3, pp. 1104-1105, 1999). Both the Sayah and Rangsten references, disclose using acid cleaning techniques. Another article, H. Nakanishi, T. Nishimoto, M. Kani, T. Saitoh, R. Nakamura, T. Yoshida, S. Shoji, "Condition Optimization, Reliability Evaluation of SiO2-SiO2 HF Bonding and Its Application for UV Detection Micro Flow Cell," Sensors and Actuators, V. 83, pp. 136-141, 2000, discloses low-temperature bonding of fused SiO_2 by first contacting the bonding surfaces with hydrofluoric acid.

[00024] According to one embodiment of the invention, functional groups are provided on opposing surfaces of the articles to be bonded. No adhesives, high temperature pre-treatment or caustic hydrofluoric acid treatments are required prior to bonding the opposing surfaces. In one such embodiment of the invention, a surface treatment of a high pH base solution such as sodium hydroxide, potassium hydroxide or ammonium hydroxide is utilized to provide functional groups on the bonding surfaces of the articles. In a preferred embodiment, the surfaces are first cleaned using a detergent followed by rinsing with an acid solution such as a nitric acid solution to remove particulate contamination and soluble heavy metals respectively.

[00025] According to one another embodiment of the invention, the surfaces are contacted with a high pH solution, rinsed, pressed into contact and gradually heated to the desired temperature, preferably to a temperature less than 300° C. It is preferable to use a "clean" heat source that does not introduce contaminants or byproducts to interfere with bonding. Such heat sources include, but are not limited to, induction heating, microwave heating, radio frequency (RF) heating and electric resistance heating. To enhance bonding, it is preferred that the surfaces are flat, as determined by performing a preliminary cleaning and pressing the dried samples into contact. Resulting interference fringes can be acquired according to techniques known in the art and interpreted to determine matching flatness. Also, an optical

flat or interferometer can be used to evaluate individual surface flatness.

[00026] Preferably, the bonding process of the present invention consists of machining each surface to be sealed to an appropriate flatness. Particularly preferred flatness levels are less than about 1 micron and roughness levels of less than about 2.0 nm RMS. After polishing, each surface is preferably cleaned with an appropriate cleaning solution such as a detergent, soaked in a low pH acidic solution, and soaked in a high pH basic solution to generate a clean surface with silicic acid-like (i.e., ≡Si-OH, =Si-$(OH)_2$, -Si-$(OH)_3$ and -O-Si-$(OH)_3$) terminated surface groups. In a preferred embodiment, the surfaces are assembled without drying. A low to moderate load (as low as 1 PSI) is then applied as the surfaces are heated to less than 300° C, for example, between 100-200°C, so that absorbed water evaporates and silicic acid-like surface groups condense to form a covalently-bonded interface.

[00027] According to an embodiment of the invention, as noted above, it is desirable to provide bonding surfaces that are flat. It is preferred to have surfaces finished to 5 micron flatness or better, and preferably 1 micron flatness or better, on the surfaces to be bonded.

[00028] For glass surfaces having a high percentage of silica, higher temperature heating is not necessarily required to form high strength bonds. For higher silica systems,

heating below 300° C is usually sufficient to form a high strength bond. On the other hand, samples that have a lower amount of silica in the glass composition may require heating to higher temperatures to form a satisfactory bond. For example, Pyrex® glass (containing approximately 81% silica) and Polarcor™ (containing approximately 56% silica), which are borosilicate glasses may require additional heating to provide sufficient bond strength for applications requiring high bond strength. The degree of heating for different bonding surfaces and glass surfaces will depend in part on the type of surface to be bonded (e.g., a fiber or a flat surface) and the desired bond strength for a particular application.

[00029] It is expected that the methods of the present invention will provide bonding strength sufficient to withstand high temperature drawing and the tension applied to preforms during drawing. Preliminary results indicate that bonding strength of high purity fused silica exceeded 150 psi. Details on the bond strength and additional information on a preferred embodiment of chemically bonding glass surfaces may be found in copending United States patent application entitled, "Direct Bonding of Articles Containing Silicon," commonly assigned to the assignee of the present patent application and naming Robert Sabia as inventor,. However, the present invention is not limited to the chemical bonding methods disclosed in the copending patent application, and it is believed that other chemical bonding techniques, wringing

and vacuum bonding can be utilized in accordance with the present invention.

[00030] In one particular embodiment of the invention, optical fiber preforms can be bonded together prior to drawing into an optical fiber. Referring to Figs. 2a-2d, at least two optical fiber preforms 30, 40 are provided, and opposing endfaces 32, 42 of the preforms are ground and polished so that the endfaces 32 and 42 have a flatness of at least 1 micron and 2 nm RMS. The surfaces are then joined together by wringing, vacuum bonding, or chemical bonding, without using an adhesive or raising the temperature of the endfaces of the optical fiber preforms to the deformation temperature of the preform material. According to a preferred embodiment, the endfaces are contacted with a solution that provides termination groups on the endfaces 32 and 42. The endfaces may be contacted with an acid solution and/or a high pH solution. Treatment with an acid will provide hydroxyl termination groups on the endfaces of the preforms. Subsequent treatment with a solution having a pH greater than 8 will provide silicic acid-like termination groups on the surface of the endfaces. After treatment of the endfaces with a solution, the endfaces 32 and 42 are joined together shown in Fig. 2b. Thereafter, it may be desirable to heat the joined preforms together to a temperature below the softening point or deformation temperature of the preforms, e.g., below 1000° C to provide a unitary optical fiber blank 50, as shown

in Fig. 2c. The blank can then be inserted in a drawing apparatus shown in Fig. 1 to produce an optical fiber 52 as shown in Fig. 2d. Alternatively, the preform 50, can be drawn to produce a rod 54, as shown in Fig. 2e; e.g. a core-cone rod utilized as a precursor article for use in manufacture of optical fiber preforms.

[00031] In another embodiment of the invention, direct bonding can be utilized to bond other glass articles such as, bar and/or sheets and the like. Such direct bonding that does not involve heating the glass articles to the softening point of the articles to be bonded is advantageous to prevent deterioration of the optical properties by heating to the softening point. For example, as shown in Fig. 3a, according to a prior art process for drawing bars from a preform 60, a first section 62 of the preform 60 is sacrificed because a clamping or holding mechanism 61 must be attached to the first section 62 to hold the preform 60 during drawing. Similarly, a lower section 64 of the preform 60 is also sacrificed during the drawing process when the preform 60 is lowered into the heating element 63 for heating the preform for drawing. According to the present invention, and as shown in Fig. 3b, sacrificial preform sections 72 and 74 may be directly attached to the preform 70 prior to drawing. The sacrificial preform sections 72 and 74 and the preform 70 are provided with flat opposing surfaces. The opposing surfaces of sacrificial section 72 and preform 70 are brought into contact, and the holding or clamping mechanism 73 can be

attached to sacrificial section 72. Opposing sections of sacrificial section 74 and the preform 70 are also brought into contact. Sacrificial section 74 is then lowered into heating element 73, preventing the loss of material from the preform 70. In one preferred embodiment, termination groups such as hydroxyl groups or silicic acid-like groups are provided on the opposing surfaces prior to contacting the surfaces.

[00032] In another embodiment, the direct bonding techniques of the present invention can be utilized to bond opposing lateral surfaces of tubes that are subsequently drawn into a dual ferrule, which are used in connecting optical fibers. According to this embodiment, as shown in Figs. 4a-4d, pair of glass tubes 80 and 90, such as Pyrex® glass tubes are provided. Lateral surfaces 82 and 92 of the tubes 80 and 90 are ground, polished and cleaned according to the present invention. The lateral surfaces 82 and 92 are then held together and directly bonded by vacuum bonding, wringing or chemical bonding. According to a preferred embodiment, the lateral surfaces 82 and 92 are contacted with an acid such as nitric acid, and then the lateral surfaces are contacted with a high pH solution such as a solution of ammonium hydroxide. Preferably, the surfaces are held together under moderate pressure of greater than one pound per square inch and heated to form a covalent bond between the tubes 80 and 90. For Pyrex® tubes, preferably the tubes are heated to a temperature

exceeding 400° C, but lower than the softening point of Pyrex®, which is approximately 675° C. The resulting product is a dual tube 96 that can be drawn into a dual ferrule structure.

[00033] It will be apparent to those skilled in the art that various modifications and variations can be made to the present invention without departing from the spirit or scope of the invention. Thus, it is intended that the present invention cover modifications and variations of this invention provided they come within the scope of the appended claims and their equivalents.